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A New Series of Advanced-Temperature Nickel-Base Alloys

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Abstract

Nickel-base alloys that do not require vacuum melting techniques and that provide high strength properties at elevated temperatures as well as a degree of workability were developed at NASA. Both increased strength and workability were obtained by utilizing tantalum alloying additions. The strongest alloy obtained in this investigation had a nominal composition of 8 wt-% tantalum, 6 wt-% chromium, 6 wt-% aluminum, 4 wt-% tungsten, 4 wt-% molybdenum, 2.5 wt-% vanadium, 1 wt-% zirconium, 0.125 wt-% carbon, and the balance nickel. This alloy demonstrated average as-cast rupture lives of 1,200, 560 and 185 hr at 15,000 psi stress and temperatures of 1800, 1850, and 1900° F, respectively. Ultimate tensile strengths of 80,000, 54,300, and 49,200 psi were obtained at 1800, 1900, and 2000° F. These results represent substantial improvements over the strongest previously developed alloy in this series. Some workability was also demonstrated by limited rolling and forging attempts with this alloy. Buttons were rolled and reduced in thickness by 23% at 1200° F without evidence of cracking. A 29% change in the diameter of $\frac{3}{4}$ in. forging bars was obtained by drop forging at room temperature. On the basis of the evaluations conducted, the strongest tantalum-modified alloy appears to have considerable potential for a variety of advanced-temperature applications, including structural members for aerospace vehicles.

Introduction

The structural integrity as well as the performance of turbojet aircraft, rocket-powered missiles, and aerospace vehicles is vitally dependent upon the materials from which their individual components are fabricated. Specific areas of importance are turbojet engine buckets, liquid propellant rocket-motor components such as the turbopump, and various structural members of aerospace vehicles. Nickel-base alloys afford an excellent means for achieving stronger, advanced-temperature, oxidation-resistant materials for these applications.

Extensive research by many investigators has resulted in the development of a variety of nickel-base alloys for elevated-temperature applications. These include the Nimonics, the Inconel series, Guy alloy, M-252, the Udimet series, Nicrotung, and many others. Various techniques have been employed to achieve improved elevated-temperature strengths with nickel-base alloys. One innovation has been to utilize vacuum melting techniques, which have the effect of reducing impurity content in the melts, thereby generally achieving alloys with improved properties. Another procedure that has increased the elevated-temperature strength properties of certain nickel-base alloys was the addition of small quantities of boron and/or zirconium.[1,2]

The addition of prime strengthening elements such as aluminum and titanium to nickel-base alloys has also been utilized in order to form the stable, dispersed $Ni_3Al(Ti)$ intermetallic compound phase. This phase provides coherency strengthening of the matrix, acts as a barrier to metal slip under load at high temperature, and contributes its strength to that of the alloy. It has been found to be largely responsible for the high strengths exhibited in a series of Ni-Cr-Al-Ti alloys,[3] and is the basis for the high elevated-temperature strengths exhibited by several commercial nickel-base alloys.

Utilizing the large research background available in this field, an investigation was conducted at NASA with the objective of providing a series of nickel-base alloys having outstanding advanced-temperature characteristics. Freche et al.[4-6] describe the results of the initial phases of this investigation. In brief, a series of cast nickel-base alloys with satisfactory strength and oxidation characteristics at 1800° F and above was developed. A basic alloy was developed that had a composition of 8 wt-% molybdenum, 6 wt-% chromium, 6 wt-% aluminum, 1 wt-% zirconium, and the balance nickel. The effects of small additions of boron, carbon, titanium, and titanium plus carbon (made by adjusting nickel content) upon alloy properties have been described.[4] The strongest alloy described by Freche and Waters,[4] the 1.5 titanium, 0.125 carbon modification of the basic composition, demonstrated as-cast rupture lives of 380 hr at 1800° F and 107 hr at 1850° F at a 15,000-psi stress. When evaluated as a turbine blade material in a turbojet engine at a blade temperature of 1650° F (turbine inlet-gas temperature of 1800° F), this alloy compared quite favorably with other superalloys evaluated on the basis of test hours compiled without blade failure.[5]

The effects of vanadium and tungsten additives upon alloy properties are described by Freche et al.[6] The strongest alloy resulting from these studies, a 4% tungsten, 2.5% vanadium, 0.125 carbon modification of the basic alloy, demonstrated as-cast rupture lives of 768, 301, and 101 hr at 15,000 psi stress and temperatures of 1800, 1850, and 1900° F, respectively, in single tests. All of the alloys evolved in these investigations provided good impact resistance. There was no evidence of catastrophic oxidation after prolonged stress-rupture testing in air with any of these alloys.

Although each of these modifications of the basic alloy demonstrated added improvements in elevated-temperature strength, it was considered desirable to extend the usefulness of this series of alloys by providing both additional elevated-temperature strength and, if possible, some degree of workability. In order to achieve these objectives, alloying studies were continued with the strongest previously developed composition, the 4% tungsten, 2.5% vanadium, 0.125% carbon modification of the basic NASA alloy,[6] utilizing tantalum as an alloying additive. Alloys were evaluated with regard to their stress-rupture, tensile, and impact-resistance properties, as well as workability. The latter evaluation was made by rolling and forging. As in the previous investigations, melts were made by high-frequency induction heating under an argon blanket, and investment-casting techniques were employed.

Investigative Procedure

The strongest previously developed alloy (4 wt-% tungsten, 4 wt-% molybdenum, 2.5 wt-% vanadium, 6 wt-% aluminum, 6 wt-% chromium, 1 wt-% zirconium, 0.125 wt-% carbon, and the balance nickel) was modified by systematic additions of tantalum to the melts. Utilizing the nomenclature of Freche et al.,[6] this alloy is designated herein as Mo-4-W+2.5V+0.125C. Tantalum additions ranging from 4 to 12% were made, and the weight percentage of nickel was reduced by corresponding amounts. As an additional example of the nomenclature used herein, the composition resulting from the addition of 4% tantalum is referred to as alloy Mo-4-W+4Ta+2.5V+0.125C.

Purity of Raw Materials

The purities of the alloying elements used, as determined by the suppliers, were as follows: nickel, 99.95+; electrolytic chromium, 99.5+; molybdenum, 99.0+; 1100 aluminum, 99.0+; vanadium, 99.8+; tungsten, 99.9+; and tantalum, 99.7+.

Chemical Analyses

Randomly selected heats of the compositions investigated were chemically analyzed by an independent laboratory. The major loss in charging elements was that of aluminum, which, in view of its tendency to vaporize, might be expected. Aluminum content varied from 5.11 to 5.84 wt-%. Considerable variation in zirconium content was also noted, the range covered extending from 0.53 to 1.43 wt-%. However, in the majority of cases sampled, the zirconium content was between 0.83 and the 1 wt-% desired. Although 1% zirconium is specified in the nominal composition of this alloy, it was not added as a melting constituent. Instead, zirconium was picked up during the melting process from the stabilized zirconia crucibles used, and the variation in weight percentage noted was probably due to unavoidable variations in melting times which would occasionally cause the melts to be exposed to the crucibles for varying time intervals.

Casting Technique

A 50-kw, 10,000-cps water-cooled induction unit was used for melting. All melts were made in stabilized zirconia crucibles under an inert gas (commercially pure argon) blanket. The average melting time was 20 min/melt. All melts were top-poured at 3150° F ± 50° into 1600° F investment molds. The molds were then permitted to cool slowly (overnight) to room temperature before removing the investment. The lost-wax process was employed to make molds of stress-rupture and tensile bars, cylindrical bars (for forging and to make buttons for rolling), and impact bars.

Stress-Rupture Data

The results of the stress-rupture tests conducted with as-cast test bars (described by Freche and Waters[4]) at 1800, 1850, and 1900° F under a stress of 15,000 psi are shown in Figure 1. A minimum of two

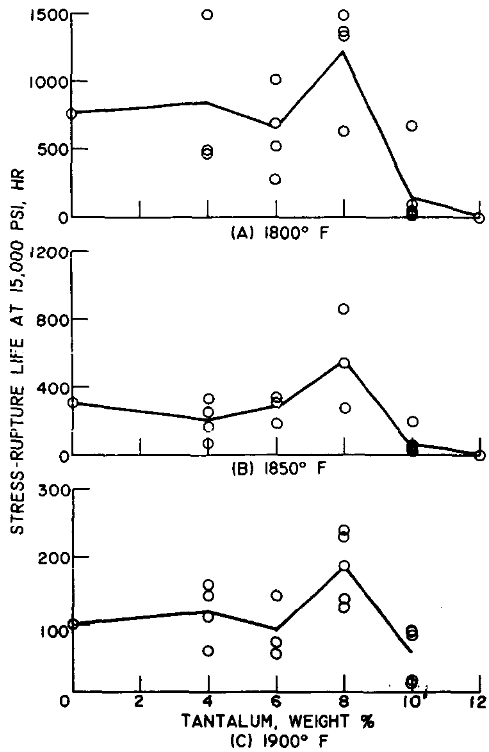


Fig. 1. Effect of tantalum additions on stress-rupture life of NASA alloy Mo-4-W + 2.5V + 0.125C at 15,000 psi stress and various temperatures. All data as cast.

and a maximum of five data points were obtained with each alloy up to the 10% tantalum modification at the temperatures cited. Interconnected straight lines drawn through the data indicate that no strengthening effect due to tantalum additions occurred up to approximately 6%. Beyond this per cent a definite increase in stress-rupture life occurred at all the test temperatures. The data show an evident peak at an 8% tantalum addition with sharply declining rupture life at 10 and 12% tantalum. On the basis of these stress-rupture data, alloy Mo-4-W+8Ta+2.5V+0.125C appears to be the strongest modification investigated. It provided average rupture lives of 1200, 560, and 185 hr at 1800, 1850, and 1900° F, respectively. It is interesting to note that the maximum life values shown in Figure 1 are substantially better than the average values cited above. All the data shown were obtained with as-cast samples, and additional increases in rupture life may possibly be obtained by suitable heat treatments.

Figure 2 provides a comparison of the as-cast stress-rupture life at 15,000 psi of the strongest alloy (Mo-4-W + 8Ta + 2.5V + 0.125C) obtained in this investigation, the strongest tungsten-vanadium-carbon modification of the basic alloy (Mo-4-W + 2.5V + 0.125C),

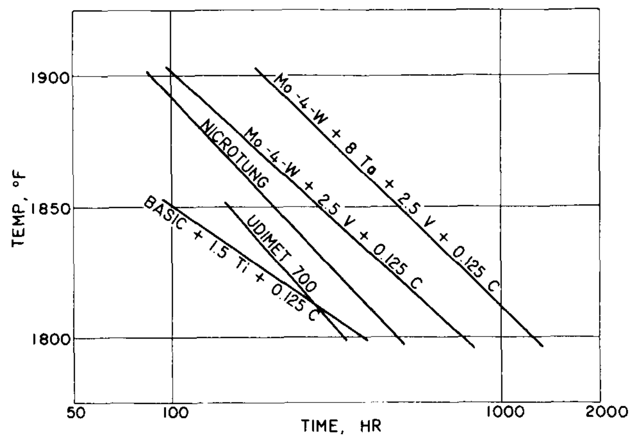


Fig. 2. Stress-rupture comparison at 15,000 psi stress of recent commercial alloys and the strongest NASA alloys. All data as cast.

and the strongest titanium-carbon modification of the basic alloy (basic + 1.5Ti + 0.125C), as well as two of the strongest commercial high-temperature nickel-base alloys. The line depicting the rupture properties of alloy Mo-4-W + 8Ta + 2.5V + 0.125C was obtained using the average life values of Figure 1. The commercial alloy data were obtained from data folders of the Westinghouse Corp. and the Kelsey-Hayes Co. It is apparent that alloy Mo-4-W + 8Ta + 2.5V + 0.125C compares favorably with the previously reported NASA alloys as well as with the commercial alloys.

It should be emphasized that this comparison is based upon limited test results for the NASA alloys. It is made only to provide an indication of the potential of the NASA alloys. If these alloys are to be considered for production, more tests must be made to provide a complete evaluation, and a casting procedure suitable for production practice must be evolved.

Tensile and Hardness Data

Tensile test data are summarized in Table I. Ultimate tensile strength, per cent elongation, and per cent reduction of area were obtained for the alloy shown to be strongest in stress rupture (alloy Mo-4-W + 8Ta + 2.5V + 0.125C) at −320°, room temperature, 1800, 1900, and 2000° F. Specimens identical to those used in stress-rupture tests were used to obtain tensile data. The as-cast values reported are averages of at least two tests. The as-rolled data, however, represent one test point obtained with a tensile specimen machined from a ¾-in. rolling bar. The latter was reduced in cross-sectional area 21.5% by rolling (2% per pas at 1200° F) prior to machining. Also included in

TABLE I
Summary of Tensile Data

Alloy	Condition	Temp., ° F	Ultimate tensile strength, psi	Elongation, %	Reduction of area, %
Mo-4-W + 8Ta + 2.5V + 0.125C	As cast	−320	129,000	7.9	19.2
		Room	134,400	5.2	15.7
		1800	80,000	5.5	8.9
		1900	54,300	9.5	12.0
		2000	49,200	7.2	11.5
	As rolled	Room[a]	155,200	3.8	8.7
Mo-4-W + 2.5V + 0.125C	As cast	Room	113,000	1.6	0.6
		1800	66,900	4.1	4.7
Basic + 1.5Ti + 0.125C	As cast	Room	106,000	0.9	3.2
		1800	56,200	4.8	5.1
Nicrotung[b]	As cast	Room	130,000	5	—
		1800	67,000	6	—
Udimet 700 (forged)[c]	Heat treated	Room	202,000	16	20
		1800	52,000	27	27

[a] Data from single test only.
[b] Data from Westinghouse preliminary data folder.
[c] Data from Kelsey-Hayes preliminary data folder.

the table for convenient comparison are tensile test data for the strongest tungsten-vanadium-carbon modification (Mo-4-W + 2.5V + 0.125C) and the strongest titanium-carbon modification of the basic alloy (basic + 1.5Ti + 0.125C) previously developed, as well as two of the strongest commercial high-temperature nickel-base alloys, Nicrotung and wrought Udimet 700. Alloy Mo-4-W + 8Ta + 2.5V + 0.125C provides approximately a 15% increase in ultimate tensile strength over alloy Mo-4-W + 2.5V + 0.125C at both room temperature and 1800° F. An increase of approximately 20% at room temperature and 30% at 1800° F was obtained over the basic + 1.5Ti + 0.125C alloy. Comparison with the commercial alloys listed shows that alloy Mo-4-W + 8Ta + 2.5V + 0.125C has a slightly higher ultimate strength at 1800° F than Nicrotung. Forged Udimet 700 demonstrated approximately 65% greater room temperature strength than the NASA alloy, although its 1800° F strength was 35% lower. The Udimet alloy also shows higher elongation and reduction in area values as might be expected from a completely wrought product.

TABLE II
Hardness Data

Alloy	Condition	Average hardness	
		R A[a]	R C[b]
Mo-4-W + 4Ta + 2.5V + 0.125C	As cast	69.5	38
Mo-4-W + 6Ta + 2.5V + 0.125C		70.2	39
Mo-4-W + 8Ta + 2.5V + 0.125C		71	41
Mo-4-W + 10Ta + 2.5V + 0.125C		72.5	44
Mo-4-W + 12Ta + 2.5V + 0.125C		73.2	45
Mo-4-W + 2.5V + 0.125C	As cast	67.9	35
Basic + 1.5Ti + 0.125C		69.6	38
Mo-4-W + 8Ta + 2.5V + 0.125C	Rolled 26% at 1200° F	76.6	51

[a] Rockwell A results are average of at least three tests.
[b] Converted from Rockwell A.

The hardness data are summarized in Table II. Rockwell C values were converted to the nearest whole number from the experimentally obtained Rockwell A values by using a standard conversion table. There is a trend of increasing hardness as tantalum content was increased from 4 to 12% for the as-cast alloys.

Impact Resistance Data

The impact resistance data are summarized in Table III. All data were obtained with a low-capacity Bell Telephone Laboratory Izod impact tester. Total capacity of the pendulum was 62.5 in.-lb., the striking velocity was 135 ips, and the specimens were $\frac{3}{16}$ by $\frac{3}{16}$ by $1\frac{1}{2}$ in. unnotched bars. When the test samples did not break, the impact resistance values were listed as being greater than 62.5 in.-lb. Tantalum additions up to 8% caused no measurable change in impact resistance. However, a marked decline in impact resistance occurred

TABLE III
Summary of Impact Data

Alloy	As-cast, room-temperature impact resistance, in.-lb
Mo-4-W + 4Ta + 2.5V + 0.125C	>62.5, >62.5, >62.5, >62.5
Mo-4-W + 6Ta + 2.5V + 0.125C	>62.5, >62.5, >62.5, >62.5, >62.5
Mo-4-W + 8Ta + 2.5V + 0.125C	>62.5, >62.5, >62.5, >62.5, >62.5, >62.5
Mo-4-W + 10Ta + 2.5V + 0.125C	>62.5, 31, 26, >62.5
Mo-4-W + 12Ta + 2.5V + 0.125C	12.5, 12
Mo-4-W + 2.5V + 0.125C	>62.5, >62.5
Basic + 1.5Ti + 0.125C	>62.5, 45, 40.2, 29, 36.5, 41.7
Nicrotung	>62.5, >62.5

as tantalum content was increased above 8%. It appears that tantalum additions above 8% are detrimental from the standpoint of impact resistance as well as rupture life, as indicated by Figure 1. It should be noted that the strongest alloy (Mo-4-W + 8Ta + 2.5V + 0.125C) also shows considerable improvement in impact resistance over the strongest titanium and carbon modification of the basic NASA alloy (basic + 1.5Ti + 0.125C), which performed quite favorably as a turbine blade material in a full-scale turbojet engine test,[5] an environment where good impact resistance is required.

Workability

Rolling Data

Buttons of alloy Mo-4-W + 8Ta + 2.5V + 0.125C, 0.20 in. thick and $\frac{3}{4}$ in. in diameter were rolled at room temperature, 1200, and 2100° F.

The faces of all buttons were machine-ground. Three methods of reduction were employed at each temperature. The first of these provided a 1% reduction from the initial thickness per pass. Another provided a 2% reduction for each of the first two passes, followed by a 1% reduction per pass thereafter. The final method employed a 4% reduction per pass. A stress-relieving heat treatment of 1 hr at 2150° F in argon followed by furnace cooling to room temperature was used after every 4% reduction regardless of how it was obtained. Prior to each stress relief, the samples were Zyglo-inspected for cracks. Rolling speed was 84 fpm, and the roll diameter was 2.5 in. The results of the rolling investigation are shown in Figure 3. Data were obtained for two rolling specimens at each condition considered. The symbol × in Figure 3 indicates the per cent reduction where cracks were first observed. The dashed lines after this symbol indicate that rolling was continued after cracking. The data indicate that the alloy has sufficient ductility to permit fairly substantial reductions in thickness of the rolling samples. At room temperature, reductions up to 12% were obtained before edge cracks were observed. At both 1200 and 2100° F, more substantial reductions were achieved. The maximum reduction obtained without observing cracks was 23% at 1200° F. From these data it also appears that a very small reduction per pass is

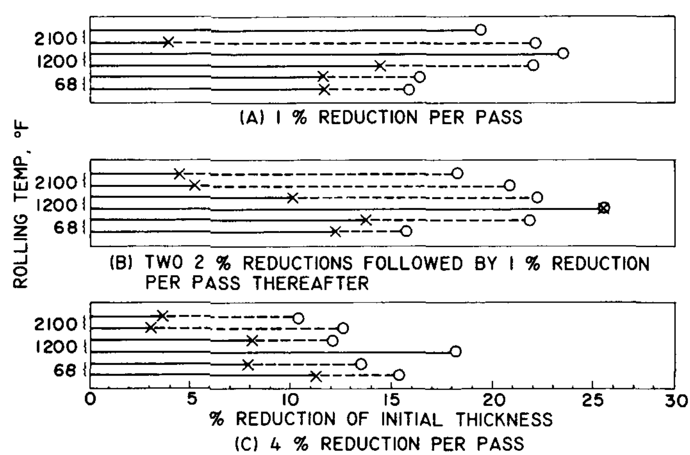


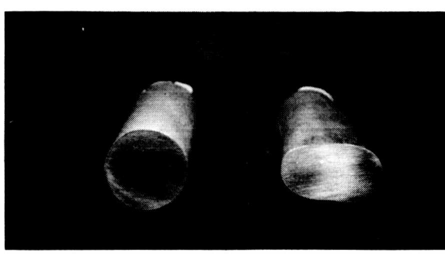
Fig. 3. Results of rolling specimens using varying percentage reductions per pass at three temperatures. (×) Point at which cracks were initially detected, (- -) continuing reduction, (O) removed from test.

more desirable than larger reductions, insofar as prevention of cracks is concerned.

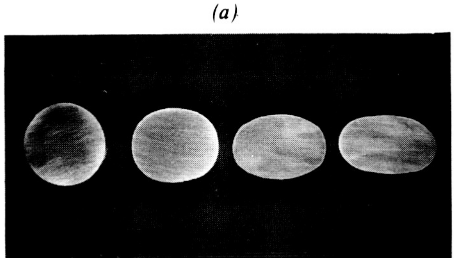
Additional rolling data of a preliminary nature have also been obtained with trimmed cast sheets of this alloy measuring 2 by $\frac{3}{4}$ by $\frac{1}{16}$ in. Utilizing reductions of approximately 0.002 in./pass at room temperature, and annealing after every four passes, several such sheets were successfully reduced more than 50% to a thickness of 0.027 in.

Forging Data

Limited attempts were made to forge alloy Mo-4-W + 8Ta + 2.5V + 0.125C at room temperature. Forging was done with as-cast vapor-blasted cylindrical rods ($\frac{3}{4}$ in. diam by 6 in. long) in a 4500-lb drop forge. The rods were placed on flat mild-steel dies, and the movable head of the forge dropped upon them from a height of 2 to 9 in. No stress-relieving treatments were attempted after any of the reductions. Figure 4*a* shows an entire forging bar before and after forging; Figure 4*b* shows cross sections cut from forging bars after various reductions.



(a)



(b)

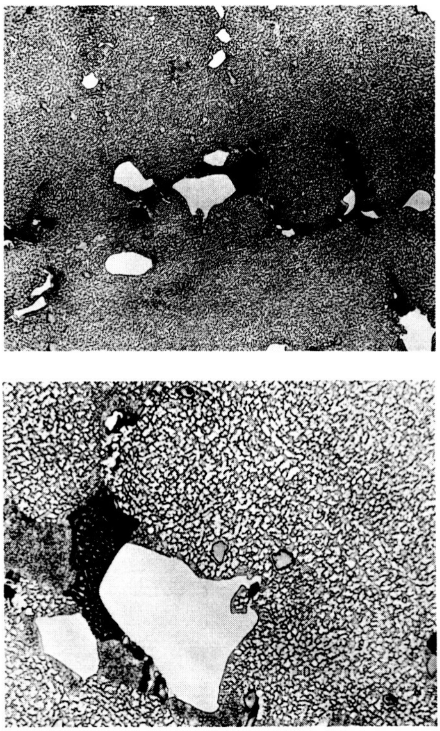
Fig. 4. Alloy Mo-4-W + 8Ta + 2.5V + 0.125C before and after various stages of forging. Maximum reduction in diameter shown, 29%.

No cracks were noted. The maximum diameter change achieved in the $\frac{3}{4}$ in. forging bars was approximately 29%. Forging attempts were discontinued when the dies began to deform.

It is recognized that more conventional and detailed forging procedures must be employed in order to evaluate fully the forging characteristics of this alloy. However, the degree of deformation obtained without cracking using the simplified procedure described tends to indicate that this alloy has a workability potential.

Metallography

Photomicrographs of alloy Mo-4-W + 8Ta + 2.5V + 0.125C are shown in Figure 5. A fine dispersion of particles is evident through-



1 inch

Fig. 5. As-cast microstructure of alloy Mo-4-W + 8Ta + 2.5V + 0.125C. Top: 125 ×. Bottom: 375 ×.

out the matrix. Preliminary electron-diffraction data indicate the presence of the Ni_3Al intermetallic compound phase, tantalum carbide, and molybdenum carbide. It can reasonably be postulated that an intermetallic phase (nickel-aluminum-vanadium) analogous to the gamma prime phase ($Ni_3(Al, Ti)$) as well as complex carbides are present.

Concluding Remarks

Much additional information could be obtained in order to describe fully the properties of the latest alloy in this series. Phase identification and workability characteristics, especially, require further delineation. However, the mechanical properties of the 8% tantalum-modified alloy compare quite favorably with commercial high-temperature nickel-base alloys and are much better than those of the other alloys in this series. As described by Freche et al.[5] turbojet-engine tests conducted at the NASA using blades of one of the earlier alloys developed in this series (basic + 1.5Ti + 0.125C) showed that a turbine blade life of 407 hr could be obtained at a turbine inlet gas temperature of 1800° F. The far superior stress-rupture and impact properties of the 8% tantalum-modified alloy indicate that it may perform even more favorably as a turbine blade material at such elevated temperatures. Finally, it would appear that the high temperature strength together with the degree of workability demonstrated with this alloy would augment its potential for use as a structural component in a variety of advanced-temperature applications. In this regard the aerospace field affords one of the most interesting immediate applications, namely, that of space vehicles which are subjected to varying temperatures above 2000° F during re-entry to the earth's atmosphere on their external surfaces, and to temperatures only slightly below this value in some of their internal structural components.

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